LAID-OPEN PATENT SPECIFICATION 1 668 347

FEDERAL REPUBLIC OF GERMANY

GERMAN PATENT OFFICE

Int.Cl.: C08 b11/20
German Cl.: 39b 1/11/20

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File Number: P 16 68 347.9 (K 64257)

Date of application: December 20, 1967

Laid open for public inspection on: September 16, 1971

TITLE: PROCESS FOR CLEANING RAW HYDROXYETHYL CELLULOSE

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K 1806 December 18, 1967

Description of the application filed by KALLE AKTIENGESELLSCHAFT for a patent for a

PROCESS FOR CLEANING RAW HYDROXYETHYL CELLULOSE

The invention relates to a process for cleaning a crude product that is collected in the production of water-

soluble hydroxyethyl cellulose and contains alkali hydroxide, in particular sodium hydroxide.

Hydroxyethyl cellulose is produced by reacting alkali cellulose with ethylene oxide. A product is obtained which contains alkali hydroxide, mostly sodium hydroxide. After the produced hydroxyethyl cellulose has been rendered soluble in water, the alkali hydroxide is removed with a mixture of organic solvents after the alkali hydroxide has been first converted into a salt by the addition of an organic acid, for example into the acetate by adding acetic acid. In the recovery of the solvent mixture used for washing out the alkali hydroxide, which is necessary for reasons of economy, the alkali salt of the respective organic acid is collected as a waste product.

In another known process, the hydroxyethyl cellulose contained in the raw product is crosslinked with glyoxalic acid so as to make it insoluble in water, and washing is subsequently carried out with cold water while maintaining a pH of from 2 to 6.

According to a third known process, neutralizing is carried out with propionic acid, benzoic acid or nitric

acid, or with a mixture of acetic acid and nitric acid, and washing is subsequently carried out with a 70% to 90% aqueous solution of isopropyl alcohol, secondary or tertiary butyl alcohol, acetone, or dioxane (whereby an aqueous acetone or dioxane solution is not used if the neutralization is carried out with propionic acid).

It is necessary in the first-mentioned known process to eliminate the waste product, which contaminates the waste water in that salts of organic acids are the cause of the high biological oxygen demand of the waste water, i.e. a so-called high "BSB" value [BSB = biologischer Sauerstoffbedarf, or biological oxygen demand. The second-mentioned process leaves behind either a slightly acid, often only gradually soluble product that has an increased viscosity value because it remains partially crosslinked, or entails a not inconsiderable loss of hydroxyethyl cellulose if said drawbacks are to be avoided. This process, furthermore, is rather closely linked to maintaining defined washout temperatures and washout times. Said process has the additional drawback that it requires that the cellulose ether be dried twice. The third-mentioned process, with the exception of cases where nitric acid is used as the neutralizer, has the same drawback as

the first-mentioned process, which is that it strongly contaminates the waste water. All of said processes have in common that the entire mass of the raw product has to be intensively mixed before it is washed out with an added reactant, which requires much expenditure in terms of energy and reduces the volume/time yield. Furthermore, said known processes have the drawback that if a highly corrosive acid is employed such as, for example nitric acid, the entire amount of acid required for the neutralization has to be filled into an agitating apparatus at once, so that the latter is subjected to such corrosion. Furthermore, many of the acids used for the neutralization such as, for example nitric acid, can be used for technical reasons only in a form diluted with water, which means that a certain amount of water in introduced into the raw hydroxyethyl cellulose, and that the raw cellulose ether accordingly has to be dried more extensively commensurate with that amount if lumping of the cellulose ether is to be positively excluded.

The task of the invention was to make available for raw hydroxyethyl cellulose a cleaning process that can be carried out in a simple way, leaving behind a waste which

contaminates the waste water to a lesser degree than salts of organic acids.

The process as defined by the invention is based on the known process for cleaning a raw product that is obtained in the production of water-soluble hydroxyethyl cellulose and contains alkali hydroxide, by neutralizing the alkali hydroxide and washing out the raw product with a mixture of organic solvents containing a methanol and, if need be, acetone, with recovery of the solvent mixture by distilling off the washing liquid, said process being characterized in that the washout step is carried out prior to the neutralizing step and with the use of a mixture consisting of

50 to 70% by volume methanol and

50 to 30% by volume acetone or isopropanol;

that the washing liquid is neutralized with phosphoric acid, nitric acid, hydrochloric acid or sulfuric acid; and that the neutralized washing liquid is subjected to distilling.

The process as defined by the invention is based on the surprising finding that hydroxyethyl cellulose, with

the specified composition of the washing liquid, is practically insoluble in a methyl-alcoholic alkali lye, and that the raw product collected in the production of the hydroxyethyl cellulose can be washed out for that reason with a washing liquid containing a great amount of methyl alcohol without neutralizing the raw product. This makes the process inexpensive and simple and makes it possible to collect a waste salt that contaminates waste water to a relatively insignificant extent. The process is even advantageous if it is applied in such a way that the alkali hydroxide is thereby only partly removed from the raw product. The contamination of the waste water is avoided in a relatively simple way in any case. A residual content of alkali hydroxide, if any, can be neutralized, if need be, in the known way, and may remain in the hydroxy cellulose, or it can be removed in the known manner.

The process reduces in a cost-saving manner the expenditures entailed by the need to remove the washed-out alkali content. Furthermore, the process avoids or reduces the corrosion of the mixing apparatus, and avoids or reduces the introduction of water into the cellulose ether. The process operates practically free of loss; it leaves

the hydroxyethyl cellulose unchanged; and requires no wasteful additions such as glyoxalic acid.

Example:

Raw hydroxyethyl cellulose produced from 100 grams cellulose, with a content of 31.5 g sodium hydroxide, was washed 4 times at room temperature, using each time 2 liters of a mixture that consisted of 50 parts by volume methanol and 50 parts by volume acetone. The amounts of sodium hydroxide removed with the washing liquids are specified in the following table:

Washing liquid 1	16.12 g NaOH
Washing liquid 2	6.76 g NaOH
Washing liquid 3	3.00 g NaOH
Washing liquid 4	1.84 g NaOH
Total	27.72 g NaOH.

Accordingly, by washing the raw product four times, 88% of the sodium hydroxide originally contained in the raw product was removed.

The residual content of sodium hydroxide was neutralized in one case with acetic acid and the sodium

acetate formed was washed out according to the known method.

The salt-containing sumps that remained in the residue after the solvent had been removed from all washing liquids used, caused contamination of the waste water which, per kilogram hydroxyethyl cellulose, expressed by the conventional "BOD" (= biological oxygen demand) (value after five days), amounted to 230 g.

When the residual sodium hydroxide content was neutralized with nitric acid, the total amount of waste water contamination came to only 170 g BOD.

Conversely, when the entire alkali hydroxide content of 31.5 g NaOH per 100 g cellulose was neutralized with glacial acetic acid before the raw hydroxyethyl cellulose was washed out, the salt sump left after the washout resulted in a waste water contamination of 450 to 470 g BOD.

Claim:

A process for cleaning a raw product containing alkali hydroxide collected in the production of water-soluble hydroxyethyl cellulose, by neutralizing the alkali hydroxide and washing out the raw product with a mixture of organic solvents containing a methanol and, if need be, acetone, with recovery of the solvent mixture by distilling off the latter from the washing liquid, characterized in that the washout is carried out prior to the neutralization, using a mixture consisting of

50 to 70% by vol. methanol, and

30 to 50% by vol. acetone or isopropanol;

that the washing liquid is neutralized with phosphoric acid, nitric acid, hydrochloric acid, or sulfuric acid; and the neutralized washing liquid is subjected to distilling.